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13. ABSTRACT (Maximum 200 words)  In this reporting period data for the physicochemical properties of the Pt/Solid polymer electrolyte interface were obtained using cyclic voltammetry and potential pulse techniques. The oxygen diffusion coefficient in Nafion was determined to be $7.4 \cdot 10^{-7} \text{cm}^2/\text{s}$ .  Several single cells were assembled and their performance at different current densities tested. The maximum power density achieved was $1.4 \text{W}/\text{cm}^2$ with a $50 \mu\text{m}$ thick membrane and electrodes with high Pt loading at $90^\circ\text{C}$ and pressure of 5atm.  A design for a fuel cell stack is proposed meeting the requirements for $7 \text{W}/\text{kg}$ specific power density. The current collectors are from gold plated titanium. High flow rate of oxygen will be used in order to achieve high heat transfer rate between the stack and the gas flow as well as removal of the product water. <i>STJ</i>				
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REPORT

**LIGHTWEIGHT SOLID POLYMER ELECTROLYTE FUEL CELL WITH  
STACK POWER DENSITY OF 3 kW/lb (7kW/kg)**

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## **1. OBJECTIVES**

The objectives of the solid polymer electrolyte fuel cell project are:

- i) Investigation of the physicochemical properties of some solid polymer electrolytes and of electrode kinetics at Pt/solid polymer electrolyte interfaces;
- ii) Optimization of low and high Pt loading gas diffusion electrode structures, membrane thickness, and electrode-membrane bonding to obtain the most effective electrocatalyst-membrane interface and hence attaining maximum fuel cell performance; and
- iii) Mathematical modeling of electrochemical, mass transfer and heat generation characteristics during cell operation to optimize fuel cell performance, with the aim of producing a stack capable of power density of 7kW/kg.

## **2. TASKS**

The following tasks have been, and are being, pursued to attain the objectives of the DARPA/ONR funded project:

**PRELIMINARY TASK: Design and Construction of Solid Polymer Electrolyte Fuel Cell Test Station**

- TASK 1. Investigation of Physiochemical Properties of Solid Polymer Electrolyte Membranes and of Electrode Kinetics at Pt/Solid Polymer Electrolyte Interfaces**
- TASK 2. Optimization of Electrode Structure and Solid Polymer Electrolyte Electrocatalyst Interface; and**
- TASK 3. Mathematical Modeling to Optimize Fuel Cell Performance**

The detailed task descriptions were presented in the proposal and first quarterly report submitted to DARPA/ONR.

### 3. WORK CARRIED OUT DURING REPORTING PERIOD

#### 3.1 TASK 1. Investigation of Physiochemical Properties of Solid Polymer Electrolyte Membranes and of Electrode Kinetic Parameters at Pt/Solid Polymer Electrolyte Interfaces

##### (i) Background

The previous report described the rationale for carrying out fundamental investigations of the platinum / solid polymer electrolyte interface, in the absence of added electrolyte. A novel electrochemical cell designed, fabricated and used for these studies was described.

The technique of cyclic voltammetry was used to check the cleanliness of the solid polymer electrolyte membrane, Nafion®. A cleaning procedure was developed to ensure that the surface electrochemical processes on platinum were clearly resolved (1). The sharpness of the oxide reduction peak, the oxide formation region, and the resolution of the hydrogen adsorption-desorption regions (Figure 3.1-1) were used as criteria to determine the efficacy of the cleaning procedure.

The cyclic voltammogram at high scan rates (100 mV/s) was also used to determine the electrochemically active area of the platinum working electrode (2). This area was then used to determine the roughness factor.

Low scan rate voltammograms were obtained at 2 mV/s between the potentials of 1.1 V (versus dynamic hydrogen electrode), where no oxygen reduction occurred, and a potential of 0.4 V (vs.DHE), where the current at the microelectrode was diffusion controlled. The cyclic voltammograms (CVs) shown in Figure 3.1-3 exhibit steady state profiles. These CVs were obtained after exposing the Nafion membrane to humidified nitrogen (background) and oxygen for extended periods of time.

Low scan rate CVs (2 mV/s) were recorded at the Pt / Nafion interface (O<sub>2</sub>-equilibrated Nafion) to determine the kinetic parameters for O<sub>2</sub> reduction. The kinetic parameters of interest here, are the exchange current density and the transfer coefficient. These kinetic parameters were obtained via Tafel analysis of the slow scan CV data. The method used (3) is described below. The steady state CV was obtained under O<sub>2</sub>-saturated conditions (curve C in Figure 3.1-2). Data points from the predominantly kinetically controlled region of the voltammogram were chosen for Tafel analysis. These data were corrected for mass-transport effects by calculating the parameter  $i_L i / (i_L - i)$ , where  $i$  is the current density at any potential and  $i_L$  is the limiting current density. A typical Tafel plot [E vs  $\log\{i_L i / (i_L - i)\}$ ] of such data is shown in Figure 3.1-3. The Tafel plot shows two well-defined linear regions (correlation coefficients better than 0.98), extending over 2 orders of magnitude, combined, in current density. It is interesting to note that Tafel plots

with two linear segments showing slopes of  $-RT/F$  (-60 mV/decade) and  $-2RT/F$  (-120 mV/decade) have also been reported for the  $O_2$ -reduction reaction in perchloric and sulfuric acids (4,5). The two section Tafel plot implies the influence of different adsorption isotherms (e.g. Temkin on an oxide covered surface and Langmuir on oxide free platinum) over corresponding potential ranges (4). The exchange current density that was obtained at the Pt/Nafion interface was higher than those obtained at any other Pt-acid interface (Table 3.1-I). This may be attributed to the lack of deleterious anion adsorption effects on the electrode in the case of the solid polymer electrolyte, Nafion. An alternative explanation is that the high proton activity of Nafion (super-acid) contributes to the better kinetics of oxygen reduction.

The oxygen diffusion coefficient and concentration were determined using the chronoamperometric method described previously (6,7). Briefly, the  $O_2$  reduction reaction is driven at the diffusion controlled rate by applying a potential step from an initial potential of 1.1 V to a final potential of 0.4 V. The time window of the chronoamperometric experiment was chosen to maintain the diffusion layer within the thickness of the membrane. A time window, spanning from 0.07 to 10 seconds, was chosen. The resulting current-time transient was recorded and the data analyzed via Cottrell plots. The quantity  $D^{1/2} C$  was obtained from the slope of the Cottrell plot; the quantity  $DC$  was obtained from the intercept. Thus, both  $D$  and  $C$  were obtained from a single experiment. The equation that was used to obtain the transport parameters,  $D$  and  $C$  for oxygen in Nafion is shown in Figure 3.1-4. The experimental current time behavior upon application of the potential step is shown in Figure 3.1-5. The current vs  $time^{(-1/2)}$  is shown in Figure 3.1-6. The values of diffusion coefficient,  $D$ , and solubility,  $C$ , are shown in Table 3.1-II. Other values of  $D$  and  $C$  obtained at other electrolyte / water immersed Pt/Nafion interfaces are also shown. Clearly the differences between the  $D$  and  $C$  values reported in Table 3.1-II are due to difference in water content (9,10). Detailed investigations of the mass transport parameters as a function of ambient humidity are in progress.

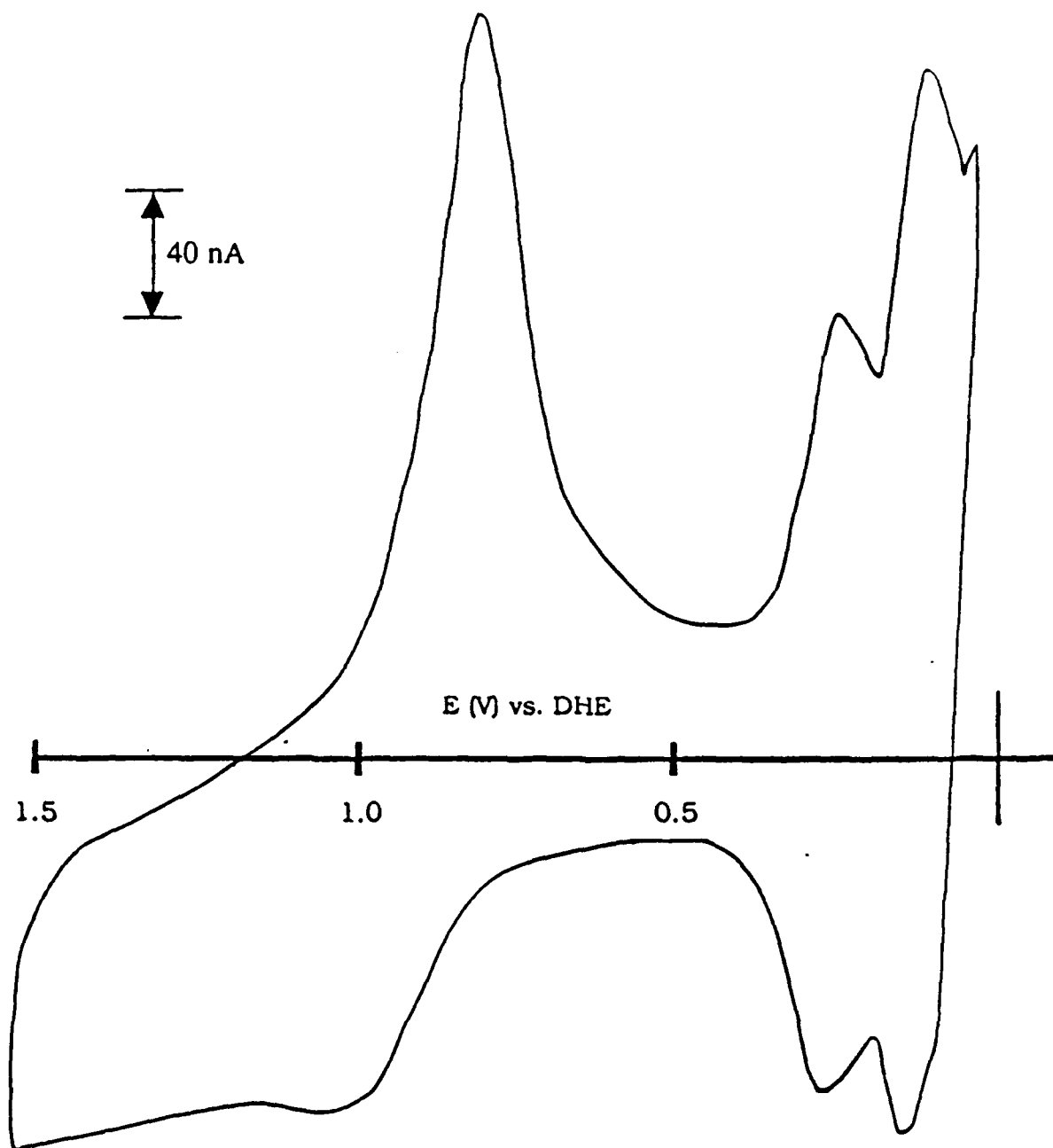


Figure 3.1.1: CV at high clamping pressure: 26 hours of cycling after Nitrogen blanketed the cell, scan rate = 100 mV/s.

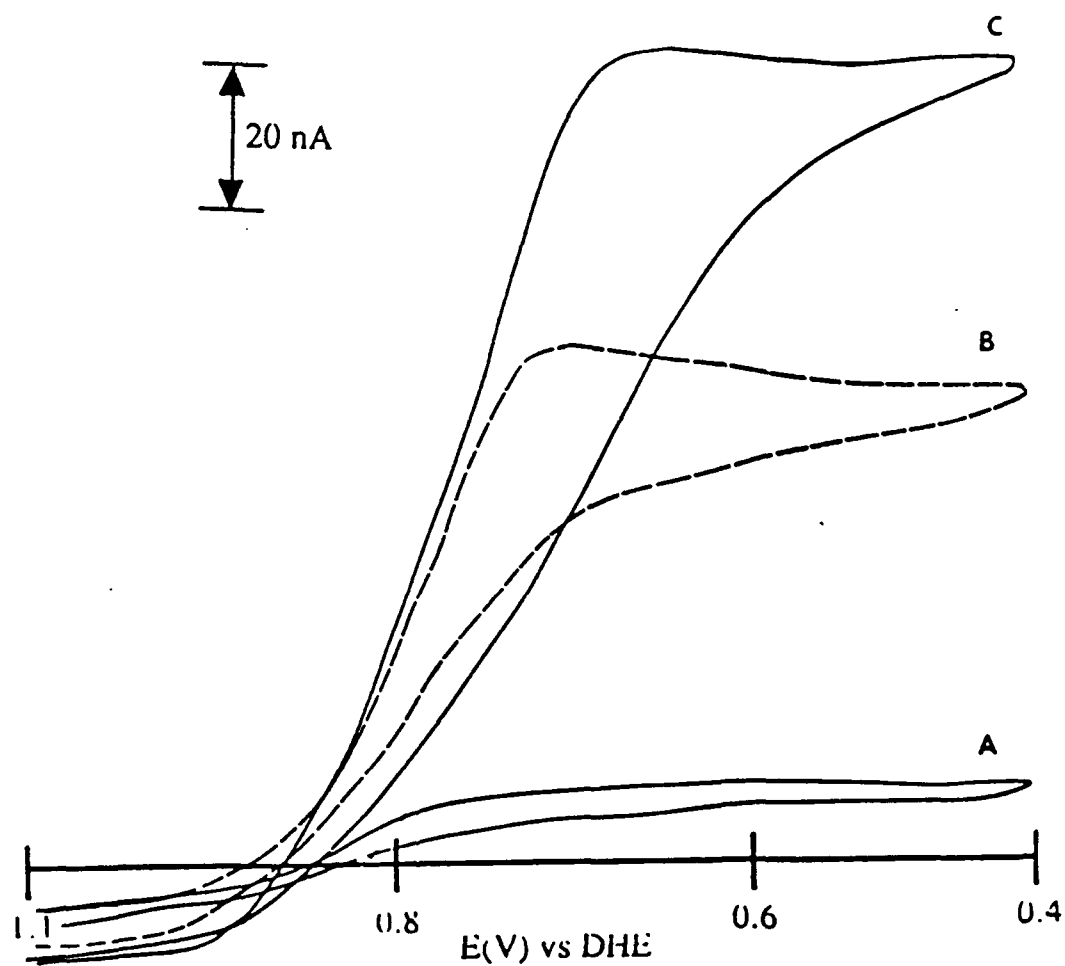


Figure 3.1.2: Slow scan (2mV/s) CVs for Oxygen reduction. (A) In nitrogen blanket; (B) in Oxygen blanket (6 hours); (C) In Oxygen blanket (26 hours).

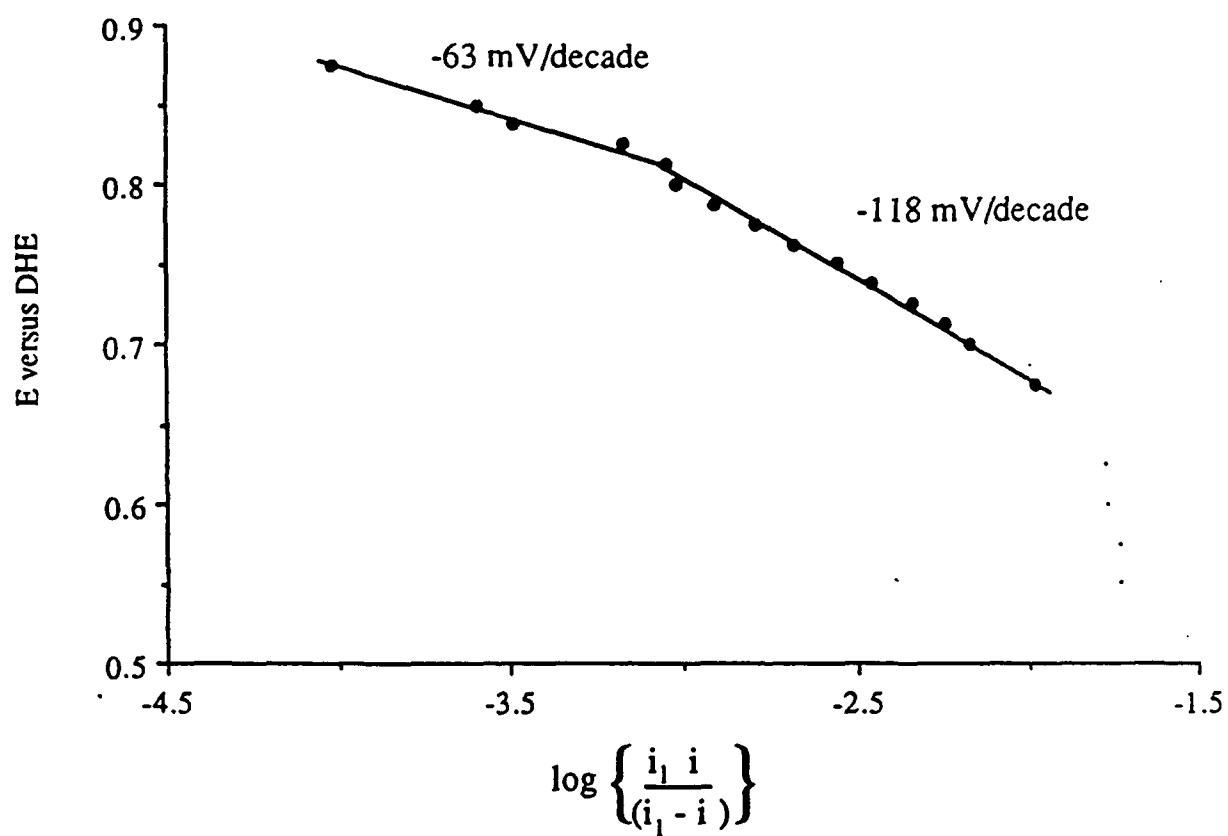


Figure 3.1.3: Mass transfer corrected Tafel plot obtained for Oxygen reduction at the Pt/ Nafion interface.



$$i = (2nFDCr\pi^{1/2}/\tau^{1/2}) + nFDCr\pi$$

$$\tau = 4Dt/r^2$$

$i$  = current (amperes)

$t$  = time (seconds)

$n$  = # of electrons transferred

$r$  = radius of the microelectrode (cm)

$F$  = Faraday constant

$D$  = Diffusion coefficient of Oxygen (cm<sup>2</sup>/s)

$C$  = Concentration of Oxygen in the film (mol/cm<sup>3</sup>)

$\tau$  = dimensionless parameter <0.8

**Figure 3.1.4:** The equation for the basis of the chronoamperometric method: simultaneous determination of  $D$  and  $C$ .

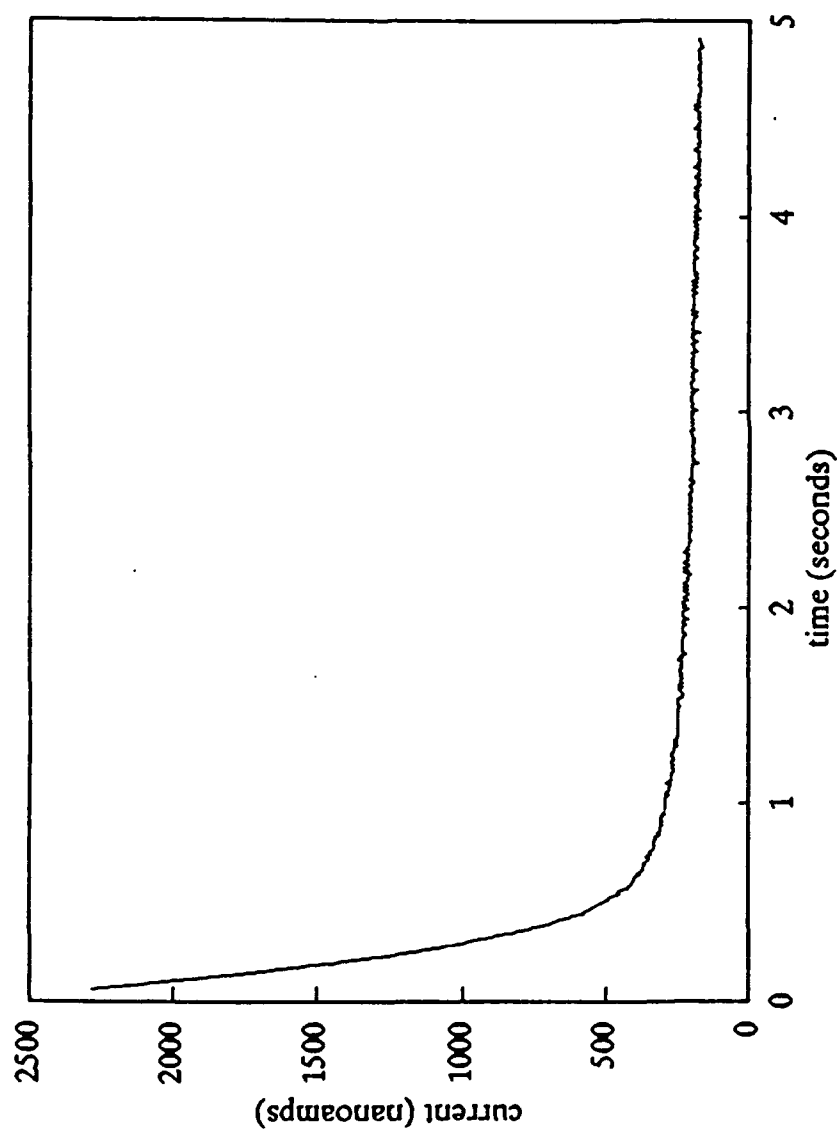


Figure 3.1.5: Current-time transient for Oxygen reduction at the Pt/Nafion interface.

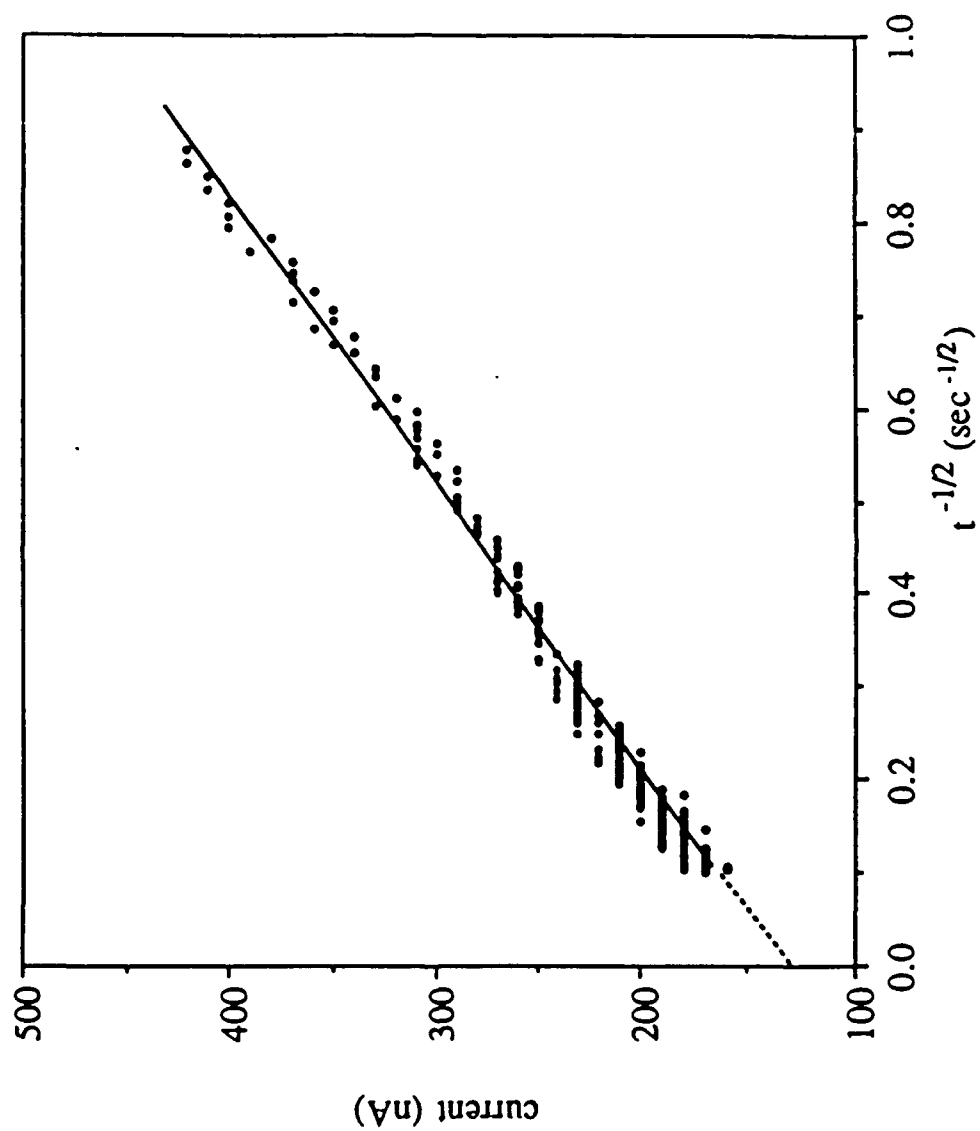


Figure 3.1.6: Cottrell plot for Oxygen reduction at the Pt/Nafion interface.

Table 3.1-I: Exchange current densities and Tafel slopes at Pt-acid interfaces

Electrolyte 25° C	Tafel slope mV/dec	Exchange current density A/cm <sup>2</sup>	Reference
Nafion membrane	-119	$7.8 \times 10^{-7}$	This work
	-63	$2.05 \times 10^{-9}$	
6.0 M TFMSA	-61	$1.4 \times 10^{-8}$	(11)
0.1 N HClO <sub>4</sub>	-60	$8 \times 10^{-11}$	(4)

Table 3.1-II: Diffusion coefficients and solubilities of oxygen in Nafion under various experimental conditions.

Parameter	Nafion solid polymer electrolyte (this work) <sup>a</sup>	Nafion immersed in water <sup>b</sup>	Nafion in H <sub>3</sub> PO <sub>4</sub> <sup>c</sup>
Diffusion coefficient, D cm <sup>2</sup> /s	$7.4 (\pm 0.3) \times 10^{-7}$	$2.4 \times 10^{-7}$	$2 \times 10^{-6}$
Concentration, C mM	26(±1)	7.2	3.8

a. Temp. = 25°C

b. Temp. = 30°C, ref. 8

c. ref. 6

### 3.2 Task 2. Investigation on Solid Polymer Electrolyte-Catalyst Interface

During this reporting period, several membrane and electrode (M&E) assemblies were fabricated and their performance under different operating conditions evaluated in single cells. The potential vs. current density behavior for these single cells assembled with membranes from different manufacturers and/or different electrodes is discussed in the following sub-sections.

#### 3.2.1. Cells with Electrochem Inc. Electrodes and Nafion Polymer Membrane

The electrodes used in these cells were provided by Electrochem Inc. The Membrane and Electrode(M&E) Assemblies were assembled in the manner described in quarterly report #5. The membrane used in these cells was Nafion, with equivalent weight 1100 and thicknesses 50 and 175  $\mu\text{m}$ . Fig. 3.2-1 present the performance of a cell with high platinum loaded electrodes(20mg/cm<sup>2</sup>) and 175 $\mu\text{m}$  thick membrane. The performance of the cells at high current densities improves significantly with the decrease of membrane thickness reaching 1.4 W/cm<sup>2</sup> with the 50  $\mu\text{m}$  thick Nafion membrane, fig.3.2-2.

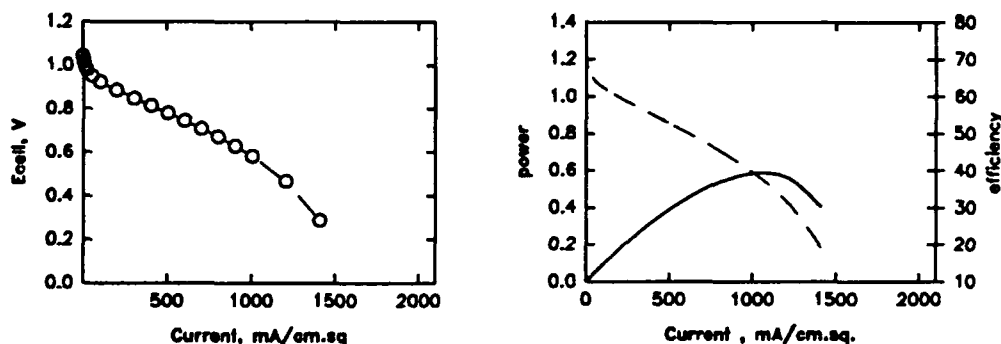


Fig.3.2-1 Electrochem Inc. electrodes, 10mg/cm<sup>2</sup> Pt loading on Carbon cloth.  
Membrane Nafion, 175 $\mu\text{m}$  thick. Pressure 5atm. Temperature 95 °C.  
Reductant oxygen.

The cell performance with different membrane thickness is shown on fig.3.2-3. One can see that the thickness of the membrane is an important parameter for the achievement of high power densities.

Another important parameter for the optimum performance of the fuel cell at high current densities is the transport of gases to the reaction zone and the removal of the

product water from the cell. Experiments were carried out for the optimization of the humidification conditions in order to obtain better performance of the fuel cell at high current densities.

The power drawn from these cells has a maximum at cell voltage of 0.5 V and the achieved efficiency is 40 to 50% calculated from the heating energy of the hydrogen-oxygen reaction.

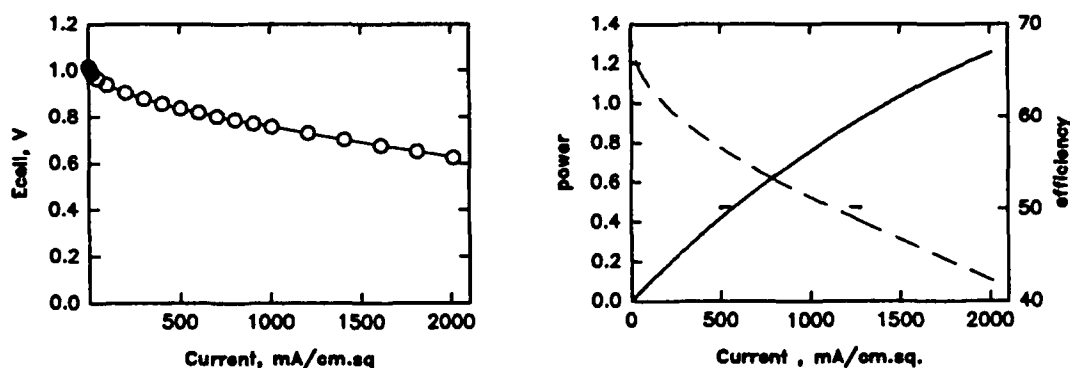


Fig. 3.2-2 Electrochem Inc. Electrodes 10mg/cm<sup>2</sup> Pt loading on Carbon cloth. Membrane Nafion 50 $\mu$ m thick. Pressure 5atm, Temperature 95 C. Reductant oxygen.

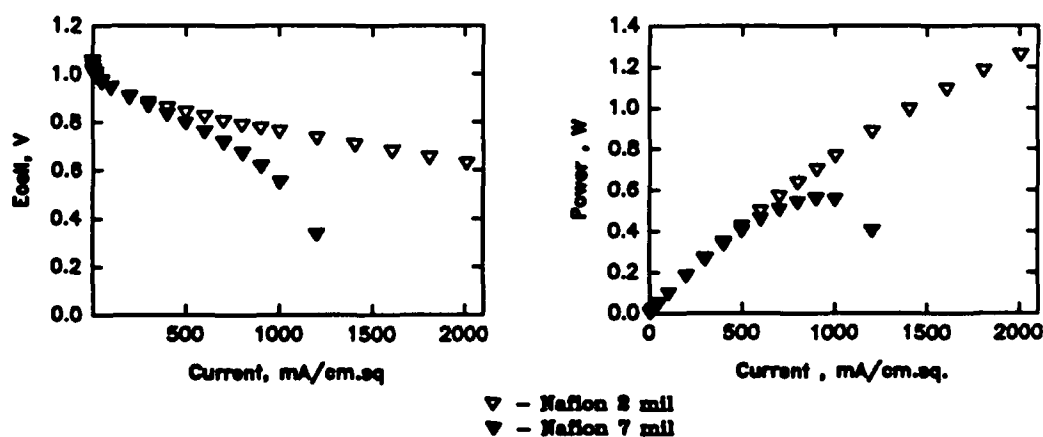


Fig. 3.2-3. Comparison between cell with 50  $\mu$ m thick membrane and one with 175 $\mu$ m thick membrane. Pressure 5atm. Temperature 95 C. Reductant oxygen.

The maximum power density achieved in our studies was  $1.4 \text{ W/cm}^2$  at current density of  $2 \text{ A/cm}^2$  and cell voltage  $E_{\text{cell}} = 0.7 \text{ V}$ . In order to achieve higher power densities more work is needed toward improvement of the characteristics of the ion-selective membrane and optimization of the operating conditions.

### **3.3 Task 3 Mathematical Modeling of Thermal and Water Management.**

The internal thermal and water management of a fuel cell are inherently interrelated. When the fuel cell is operating, water is being produced at the cathode as a result of the electrochemical reaction while heat is produced due to irreversibilities. The water production rate, ( $\text{g/cm}^2$ ) varies with fuel consumed while the thermal load is a function of fuel consumed and cell operating voltage. This means that given an excess reactant gas condition, product water increases linearly with current density ( $\text{amps/cm}^2$ ) while the thermal load increases with the square of the current density. Thus a thermal and water management system must compensate for changing operating conditions.

The performance goal of  $7 \text{ W/g}$  provides extraordinary operational problems for an SPE fuel cell. To achieve this power density the electrochemistry must be maximized while the construction materials are minimized. This study has established that the maximum expected electrochemical performance of a electrode membrane assembly is  $4 \text{ W/cm}^2$  at an individual cell voltage of  $0.5$ . This performance translates to a specific fuel cell mass density of  $0.6 \text{ g/cm}^2$  of active cell material. The bipolar plate has been identified as the critical component in achieving the lightweight necessary.

The lightweight requirement necessitate that the system that removes heat from the cell must also provide water management. Considering the enthalpy of the hydrogen-oxygen mixture the irreversibilities will be approximately 1.75 times the electrical power output from the cell. Given a maximum electrochemical performance of  $4 \text{ W/cm}^2$ , this corresponds to a specific thermal load of between  $5.25$  and  $8.75 \text{ W/cm}^2$  (for comparison the solar constant is  $0.14 \text{ W/cm}^2$ ). This extraordinary heat flux is further complicated by the low glass point temperature of the membrane material ( $140^\circ \text{ C}$ ).

A number of thermal and water management methods have been considered, including cold plate, internal evaporative cooling, and gas recirculating schemes. This study has concluded that considering the performance goal a high pressure recirculating of the oxygen with external cooling and dehumidification was optimum light weight

option. An operating pressure of 20 atmospheres was chosen to increase gas density and thus give a high volumetric specific heat.. Under this pressure condition, using todays membrane material, there would be significant gas cross over due to diffusion. Cross over results in a reduction in utilization efficiency and cell heating. The high pressure design assumes an advanced membrane capable of reducing diffusion losses.

The following two subsections describe the general goals that were set for the over all fuel cell, and the resultant bipolar plate design and gives an example fuel cell specification based on the design.

### 3.3.1 Fuel Cell Design

The fuel cell design criteria is as follows:

Specific Power	7 W/g
Reactive Gases	Pure Hydrogen and Oxygen
Voltage at 0.1 Amps/cm <sup>2</sup>	1.0
Electrolyte Ionic Resistance	0.06 $\Omega$ /cm <sup>2</sup>
Maximum Specific Cell Power	4+ W/cm <sup>2</sup> at 0.5 volts/cell, 8.4 amps/cm <sup>2</sup> of active cell area
Specific Fuel Cell Weight	0.6 g/cm <sup>2</sup> of active cell area
Maximum Membrane Temperature	140° C (3.6 Atm. Vapor Pressure)

It is assumed that the fuel cell stack will be placed in a pressurized container to reduce stress from internal reactant gas pressure. An external heat exchanger would be provided to cool and dehumidify the recirculated oxygen. The bipolar plate design is stack-able with internal reactant gas manifolding. Reactant gas connections are made at the end plates. End plates and stack compression bolts squeeze the individual cells providing sealing by gaskets and reduced electron contact resistance between the electrode and the bipolar plate current collectors.

The bipolar plate design criteria is as follows:

- Low Specific Weight (g/cm<sup>3</sup>)
- High Electrical Conductivity
- Provide an Even Distribution of reactant gases to the electrode
- Minimize Thermal Gradients across the Electrode
- Corrosion Resistant

Historically, graphite has been successfully used as a bipolar plate material. However due to its low electronic conductivity it is not suitable for the high current density condition in this study. A survey of possible materials was conducted. The



survey considered, electronic resistance, specific gravity, permeability, Youngs modulus of elasticity and yield strength. Structurally the bipolar plate must be lightweight but strong enough not to deform due to stack compression. Some of the materials that were considered are as follows:

**Bipolar Plate Materials**

Bipolar Plate Base Material	Specific Gravity	$\Omega$ cm	Modulus of Elasticity $\text{Lb/in}^2 \times 10^{-6}$	Yield Strength $\text{Lb/in}^2 \times 10^{-3}$
Graphite	2.26	$1.38 \times 10^{-3}$	0.7	
Copper	8.96	$1.67 \times 10^{-6}$	16.0	5.0
Titanium	4.54	$4.78 \times 10^{-4}$	16.8	80.0
<b>Material Coating</b>				
Platinum	21.45	$9.83 \times 10^{-6}$	21.0	
Gold	19.32	$2.19 \times 10^{-6}$	12.0	

Based on the survey, titanium was chosen as the best base material. For improved corrosion resistance and reduced contact resistance the titanium surface will be primed with a thin platinum coating followed by gold surface coat.

The choice of a metal bipolar plate eliminates water management by wicking through the bipolar plate as in the Ergenics fuel cell design<sup>1</sup>. Other fuel cell manufactures, such as Ballard<sup>2</sup> and IFC<sup>3</sup> have successfully use a recirculating reactant gas design for water and thermal management. For example the, IFC alkaline fuel cell used in the space shuttle recirculates the reactant gas through a condenser where the water is separation from gas by centrifugal pump.

The bipolar plate design uses recirculated oxygen for cooling and humidity control of the electrodes, See Figure 3.3-1. An external heat exchanger cools and dehumidifies the oxygen before it is re-injected into the fuel cell. Product water is removed from the water reserve in the condenser. On start up or a transit operating condition the water reserve can provide additional humidity to the oxygen supply. Make up oxygen is provided by a pressure regulator set to maintain the cathode pressure at 20 atmospheres. A small part of the oxygen reacts with the electrode while the rest is sensibly heated and humidified. The oxygen recirculating rate is a constant. The fuel cell temperature and internal humidity is controlled by the heat exchanger external cooling fluid loop. At a low thermal load the heat exchanger supplies warm humid

<sup>1</sup>Ergenics Power Systems, Inc. Wyckoff, N.J.

<sup>2</sup>Ballard Power Systems, North Vancouver Canada

<sup>3</sup>International Fuel Cells, South Windsor, Connecticut

oxygen to the cells, at high thermal load the oxygen supply is from the heat exchanger is cooler and drier. The pressure drop in the recirculating loop is designed to a pumping power of  $0.11 \text{ W/cm}^2$ , this represent 2.5% of the maximum gross power.

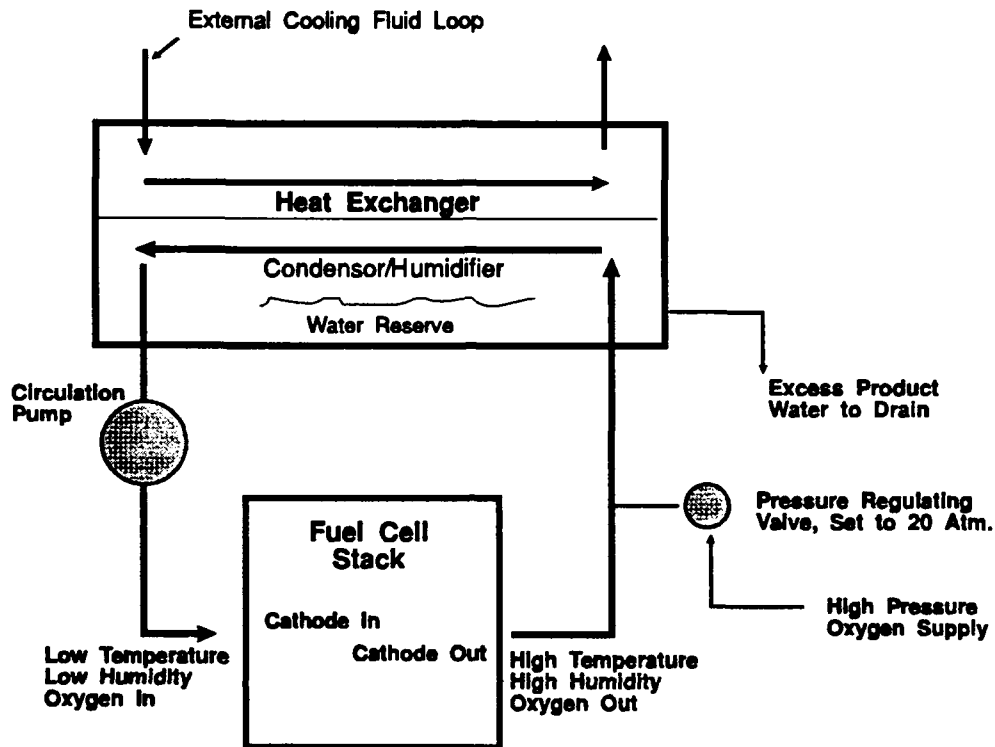


Figure 3.3-1 Recirculating Cooling System

The reactant gas flow distribution over the electrode will be critical to the successful operation of a high power fuel cell. Ideally the gases would be evenly distributed over each electrode, excess water would be removed, and there would be no temperature gradients across the electrode. A fluid boundary layer effectively insulates the electrode and thus must be kept to a minimum.

The bipolar plate design was chosen on the basis of and even distribution of oxygen across the cathode and a reduced thermal gradient. The design uses passages inside the current collector to distribute the oxygen across the electrode. The oxygen is then jetted onto the cathode surface by a series of small holes, See the bipolar plate cross section in Figure 3.3-2. The pressure drop is small being designed to approximately .25 atmospheres. This pressure drop is sufficient to ensure an even distribution of oxygen

between the multi cells in the stack. The jetted oxygen provides turbulence close to the electrode so that water vapor is removed and a fresh supply of cool oxygen is constantly bathing the cathode surface.

The bipolar plate will be constructed of two sheets of 0.015 cm thick titanium. The construction consists of stamping, punching, drilling and electric resistance welding to obtain the desired shape. The following figure shows a cross section of the bipolar plate. Detailed drawings of internal manifolding and stack design will be provided in the final report.

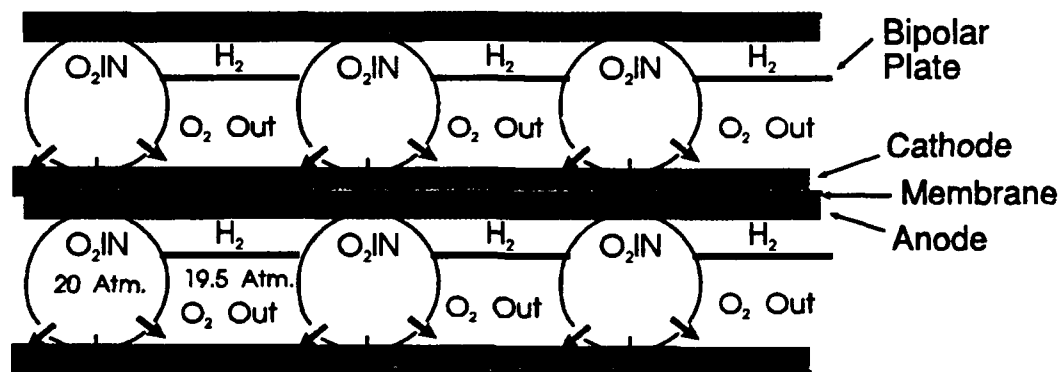


Figure 3.3-2. Bipolar Plate - Cross Sectional View

Oxygen enters the stack by passage ways in the bipolar plate. The passage ways serve as current collectors. Holes to the cathode side of the electrode are provided to allow the oxygen to be *jetted* into the reaction chamber. The holes are sized such that the pressure drop entering the reactive chamber is small but large enough to ensure an even distribution of fresh oxygen across the electrode. The drop in pressure slight lowers the temperature of the oxygen. The fluid dynamic Reynolds Number indicates that the jetted flow is turbulent. The effect of the jet action is approximately 20 times the hole diameters and is designed to agitate the surface of the cathode.

### 3.3.2 Example Fuel Cell Design

Calculations on the described fuel cell design indicate that the following SPE fuel cell specifications are possible.

Example Fuel Cell Specifications  
100 kW, 100 Volts, 1000 Amps

Active Area per cell	119 cm <sup>2</sup> 4.22 W/cm <sup>2</sup>
Individual Cell Power	500 W
Individual Cell Weight	34.9 g
Thermal Load per Cell	980 W
Cell Thickness	0.24 cm
Number of Cells	200
Stack Length <sup>4</sup>	50 cm
Stack Weight	14.306 kg
Stack Volume	7.15 liters (SG = 2.01)
Stack Power Density	6.99 kW/kg 14.06 kW/liter

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<sup>4</sup>Stack includes titanium end plates and stack compression bolts.

## **5. PUBLICATIONS**

S. Srinivasan, S. Somasundaram, D. H. Swan, H. Kock, D. J. Manko, M.A. Enayetullah and A. J. Appleby, "A Design Study of High Power Density Solid Polymer Electrolyte Fuel Cells," Proceedings of the Symposium on Fuel Cells, 89-14, 168, The Electrochemical Society, Pennington, New Jersey (1989).

## **6. PRESENTATIONS**

- (1) A. John Appleby, "A Design Study of High Power Density Solid Polymer Electrolyte Fuel Cells", by Supramaniam Srinivasan, Sriram Somasundaram, David H. Swan, Herman Koch, David J. Manko, Mohammad A. Enayetullah, and A. John Appleby, Annual meeting of American Institute of Chemical Engineers, San Francisco, CA November 6-7, 1989

## **7. VISITORS**

April 5, 1990, Mr. George Huff, Amoco Corporation, Naperville, Illinois.

April 17, 1990, Mr. Michael Ce, NASA/JSC, Houston, Texas.

April 18, 1990, Sergio Kapusta, Shell Development, Houston, Texas.

May 21, 1990, Chary Soo, Kim, Korea Institute of Energy Resources, Daejeon, Korea.

June 28, 1990, Tom Walters, Westinghouse Savannah River Lab, Aiken, South Carolina.

## **8. VISITS**

- (1) A. John Appleby, Annual Meeting of American Institute of Chemical Engineers, November 6-7, 1989, San Francisco, California
- (2) S. Srinivasan and M.A. Enayetullah, Fall Meeting of The Electrochemical Society, Hollywood, Florida October 15-20, 1989

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